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David Taylor Research Center

Bethesda, MD 20084-5000

DTRC-SME-91-43 August 1991

Ship Materials Engineering Department
Research and Development Report

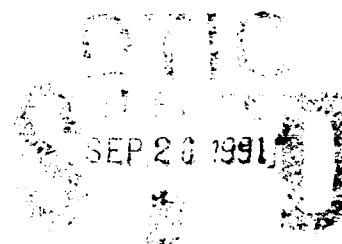
Bimetallic Tubulars Via Spray Forming

by

Paul Kelley

Angela Moran

DTRC-SME-91-43 Bimetallic Tubulars Via Spray Forming

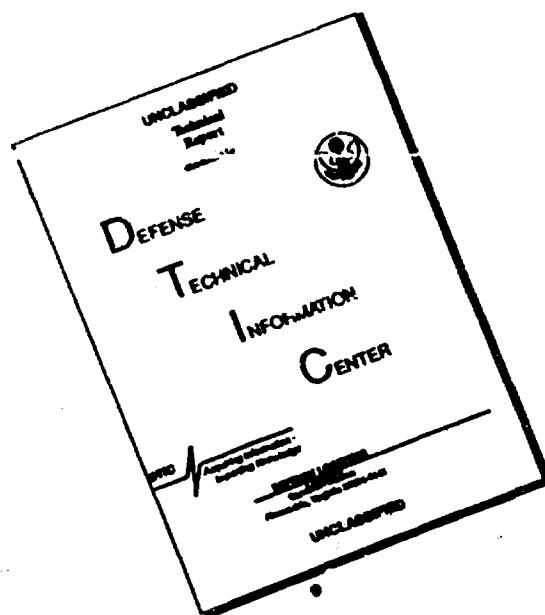


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BIMETALLIC TUBULARS VIA SPRAY FORMING

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ABSTRACT

High deposition rate spray forming is a new technology which has been developed to reduce the costs and to improve the performance of a wide variety of engineering alloys. The purpose of this investigation was to demonstrate the ability to produce layered bimetallic structures via the spray forming process. Copper tubulars were used as collectors for spray deposited alloy 625. The collectors were varied in thickness and various interfacial alloys were incorporated to promote bonding between the copper and superalloy. Microstructures and bond strength were determined.

ADMINISTRATIVE INFORMATION

This report was prepared for Aerojet TechSystems Corporation who provided independent funding under Work Unit 1-2821-888. Chester Kawashigi was the sponsor at Aerojet. This work was supervised within the Metals and Welding Division by Dr. O.P. Arora.

INTRODUCTION

In the spray deposition process, a stream of molten metal is atomized by inert gas, producing a spray of liquid droplets which are cooled by the gas and accelerated towards a substrate, where they consolidate to form a nearly fully dense deposit (Figure 1). The process improves on ingot metallurgy in that a rapidly solidified, grain-refined microstructure with limited segregation is produced. Spray forming exhibits the beneficial characteristics of powder metallurgy processing without numerous stages, such as powder production, storage

and handling, sintering and hot consolidation. A program at the David Taylor Research Center sponsored by the Office of Naval Technology evaluated the feasibility of utilizing (Osprey) spray forming to produce alloy 625 (Ni-Cr-Mo) piping. The results of this program showed that fully dense preforms could be sprayed and rolled extruded into piping with properties equivalent to conventionally processed piping at substantially reduced costs[1]. Cost savings projections for simple shapes such as piping tubulars produced via spray forming are as high as 30-50% of costs of conventional processing technology. The technology is alloy non-specific and therefore is applicable to a wide range of metal systems.

OBJECTIVE

The objective of this program was to assess the feasibility of using spray forming to manufacture a combustion chamber structural jacket and liner. The approach included determining and optimizing spray forming process parameters, manufacturing alloy 625 and copper bimetallic tubular preforms, and producing ZrCu tubular preforms for evaluation.

BACKGROUND

Bonding Techniques

The established technique for generating a bond during spray deposition is to preheat the substrate to a high percentage of the absolute solidus temperature of the spray material. In the case of alloy 625 on copper this would require that the copper be preheated above its solidus, which would cause unacceptable melting. In addition preheating would require incorporating a plasma torch into the DTRC facility and would involve significant expense. For these reasons, preheating was not considered an option.

Spray forming is typically performed at deposition rates that range from 20 to 200 kg/min and results in very high heat input rates to the liner. In the production of tubular product the size of the full-scale combustion chamber, deposition rates would be at the high end of this range and would likely involve the use of two or more nozzles. In contrast, conventional plasma techniques run at lower rates by two orders of magnitude. This high rate of deposition, the thin-walled geometry of actual ZrCu liner, and the large difference in solidus temperatures (1288°C for alloy 625, 1080°C for copper) raised concerns that the copper would melt during deposition of the structural jacket and damage the cooling passages.

An alternate technique to preheating, depicted in Figure 2, is the use of a braze interlayer applied to the liner prior to spray deposition of the jacket. The braze material would melt and bond to the jacket either during the deposition process or in a post-deposition heat treatment. Interlayer brazing is seen as a relatively simple, "low tech" method of bonding. It can be performed with a small number of processing steps and lends itself readily to a larger scale.

EXPERIMENTAL TECHNIQUES

Thermal Response Study

A study was performed to determine the effect of copper liner section thickness on its thermal response. Alloy 625 was spray deposited on three copper mandrels, all 100mm OD and about 350mm in length, but with varying wall thickness: 12mm, 3.6mm, and 1.5mm. Each liner was lightly grit blasted with

steel shot, then rinsed with acetone and isopropyl alcohol before being mounted on the manipulator. The same arrangement for mounting was used in each run and spray height was kept constant.

Processing parameters are listed in Table 1. The melt flow rate and jacket thickness were kept relatively uniform from run to run. There were variations in the resultant gas/metal ratio, although not at significant levels. After completion of deposition, the atomizing gas was left on and the preform was translated and rotated under the gas in order to minimize melting of the liner.

Interlayer Braze Study

In order to incorporate the benefits observed in melting of the copper liner during deposition, a braze interlayer was used to generate a bond. Alloy CA101 copper mandrels with 12.7mm wall were lightly grit blasted and washed with acetone and isopropyl alcohol. For the initial test, grade 1100 aluminum 0.75mm thick was sprayed onto the mandrel using conventional plasma techniques. Aluminum was chosen for its ability to form low melting point phases with copper and high melting point phases with nickel. The high melting point alloys should prevent the aluminum from migrating extensively into that material, while the low melting phases are desirable for bonding to copper during deposition. This alloy is not suitable for the final configuration because of the potential for forming brittle intermetallics, but is designed to demonstrate interlayer response. After deposition the laminate was given a diffusion heat treatment at 900°C for 5 hours in air.

A commercially available braze with a chemistry of Cu-37.5Mn-9.5Ni was also tried. It was sprayed via a high velocity oxygen fuel (HVOF) technique on a grit blasted and cleaned CA101 mandrel at a thickness of 100 microns. The HVOF technique combusts an oxygen/propylene mixture to generate a supersonic carrier jet for the metal powders. After spray deposition a section of the laminate was held at 960°C for 15 minutes in flowing hydrogen and cooled at 600°C/hour. Another section was given the same heat treatment at 1025°C for 15 minutes.

Spray Forming of ZrCu Liner

Zirconium copper feedstock was induction melted in an alumina crucible under nitrogen cover gas. A mild steel substrate 100mm OD with a 1.5mm wall was grit blasted and then mounted on the manipulator at a flight distance of 420mm. Rotation of the substrate was 240rpm. Impingement of the spray was normal to the mandrel axis (0°) or scanned towards the withdraw direction by a small amount (-1 to -6° at 16Hz). Exhaust gas temperature and preform surface temperature were measured using a thermocouple and a two-color IR pyrometer, respectively. Further details of spray parameters are listed in Table 2.

An aging study was performed on preform #113 and the ZrCu feedstock. Specimens were solution treated at 980°C for 1 hour and immediately water quenched. After aging for 1 hour at a range of temperatures and air cooling, a number of hardness measurements (>6) were taken and averaged. Samples of feedstock and preforms were evaluated in terms of chemical composition. Oxygen determination was made using a high temperature reduction process with carbon.

Mechanical Testing of ZrCu

Zirconium copper tensile specimens were machined from the outer wall section of Run #114. Specimen geometry included 0.252 inch gage diameter, 1.25 inch gage length, and shoulder radius of 0.25 inch. Before machining, the specimens were divided into five groups and heat treated as per Table 3. Each group was tested in air at room temperature as per ASTM E-8 except Group #3. This group was tested in flowing argon at 540°C (1000°F) using the same strain

rates (typically 3×10^{-5} up to yield, 10^{-4} until breakage) as the other tests. However, because no strain gage or extensometer could be used at this temperature, specimen strain was determined by correlation of crosshead travel to extensometer measurement made in previous room temperature tests. Conventional light metallography and hardness measurements were performed in the machined thread section after tensile testing at room temperature.

RESULTS AND DISCUSSION

Thermal Response Study

The study to determine the effect of thermal mass of the copper liner on its thermal response provided interesting results. The copper liner with a 1.5mm wall was completely melted by the alloy 625 jacket. The liner integrity was maintained until deposition was complete, but then the copper melted with the majority forming a pool in the ID of the jacket. Despite the unacceptable condition of complete wall melting, two potentially beneficial results were seen in the microstructure at the interface. The copper had infiltrated the deposit and filled much of the "cold" porosity in the ID material. This porosity is a result of rapid quenching of the deposit by the substrate that reduces the level of liquid in the deposit needed to fill the interstices between solidified droplets. This type of porosity can be reduced with increased melt superheat, but may be difficult to eliminate with the high thermal diffusivity of the copper substrate. If the copper completely wets the alloy 625 and fills cracks and porosity, it is believed that the inner diameter 625 material will have better mechanical properties, especially in fatigue.

Another beneficial result of the copper melting is the formation of a strong metallurgical bond. In simple hammer and chisel shear tests of the laminates, large deformation of the copper was needed to achieve bondline failure. This indicated that bond strength is approximately equal to the copper yield strength.

The liner with a wall thickness of 3.6mm only melted in the middle of the length of the test piece, while both the beginning and end were unmelted. A small pool of copper formed in the ID where complete melting of the copper had taken place, however the majority of the ID of the liner remained undeformed. At the beginning of deposition, the liner is cold and has sufficient thermal mass to prevent melting. At the end it is believed that the continuation of the atomization gas without the spray prevents melting. This change of response from the middle to the ends is reflected in the microstructures. Figure 3a is a photomicrograph from the middle of run #115, while 3b shows the poor bond at the beginning of the run. Porosity in the ID material decreases as the run progresses.

Figure 4 is a picture of the superalloy jacket formed over a 12.7mm thick copper wall. Intimate contact at the interface was seen along the length and circumference of the liner. Because solidification of the jacket takes place around a relatively cold liner, the copper is held under compressive forces. With the larger thermal mass, no melting of the copper was seen and no bond was formed.

In scaling up from the 3.6mm wall test piece to the full-sized liner the deposition rates would probably increase by a factor of 10, but the liner thermal mass will increase by more than that amount. It is believed that the liner thermal response will remain mostly unchanged in production of the actual combustion chamber and melt through of the copper liner should not be a problem. If necessary additional cooling can be obtained by circulating gas through the cooling channels during deposition.

Interlayer Braze Study

Earlier tests using silver braze on thin-walled mild steel tubulars resulted in braze material migrating completely away from the steel/alloy 625 interface. This is presumably the result of surface tension driven flow into the hot alloy 625 deposit and makes subsequent isothermal braze treatments ineffective. Although the braze was metallurgically bonded to the steel substrate, the substrate thermal mass was not sufficient to prevent braze melting during deposition. This showed the importance of having both the braze in good thermal contact with the liner and sufficient liner thermal mass to prevent braze melting during deposition. Actual formation of the brazed joint will then have to be performed in a post-deposition isothermal heat treatment.

An aluminum interlayer was deposited on a liner having large thermal mass (12.7mm wall), but because of its low liquidus temperature the aluminum did melt and migrate partially into the deposit during spraying. Energy dispersive x-ray microanalysis was used to identify the dark phase in the alloy 625 material (Figure 5) as containing aluminum and copper; both elements are not present in the starting alloy 625 feedstock. Application of aluminum was not successful in stopping migration into the deposit. However the beneficial result of reduction of cold porosity was again seen.

Alloying of the aluminum into the copper liner can also be seen. In addition there is a continuous second phase at the interface (Figure 5). It is believed that these are oxides introduced during plasma deposition of the aluminum. After heat treatment of 5 hours at 900°C the microstructure was largely unchanged with the bond strength increased slightly. Bond strengths, measured qualitatively with hammer and chisel shear tests, were only moderate with failure occurring along the oxides at the interface. With a cleaner deposition technique for the aluminum (such as low pressure plasma) much higher bond strengths are expected.

Measurements of microhardness across the interface were also taken and results plotted in Figure 6. Peak hardness is seen at the interface between the two materials and can be attributed to the presence of intermetallics or oxides. Cracks can be seen at the interface in Figure 5 and may be a result of stresses applied during rapid cooling after heat treatment.

This clearly is not an optimal system. However it did demonstrate that a low melting point material could be applied to the liner and remain at the interface and form alloys with both materials. Efforts using a conventional braze alloy (Cu-37.5Mn-9.5Ni) also failed to achieve acceptable bond strength because of oxidation during HVOF deposition. Micrographs of the interface before and after heat treatment (960°C for 15 min.) show the presence of oxides in the braze material (Figure 7). After heat treatment the small gap between the braze and alloy 625 was eliminated, indicating some flow of the braze. However, the bond was weak. Oxidation of the braze is mainly in the form of manganese oxide. Because metallic manganese is the freezing point depressant in this system, its loss raises the melting point of the braze dramatically. Brazing at a higher temperature (1025°C), however, did not improve the bond strength or change the microstructure.

Spray Forming of ZrCu Liner

Microstructures of the spray formed product and wrought feedstock are shown in Figure 8. Annealing twins are seen in the wrought material. The benefits of spray forming are evident in the photomicrograph of the as-sprayed material. It has equiaxed grains that are comparable in size to the wrought product along with a finer distribution of copper zirconium intermetallic

precipitates.

A series of preforms were made in order to obtain a ZrCu tubular with minimal ID "cold" porosity (Table 2). Scanning the spray towards the withdraw direction between -1° and -6° helped to reduce leading edge buildup and cold porosity. Decreased gas/metal ratio was also employed and resulted in higher exhaust gas temperatures, a measure of the total amount of energy removed from the metal spray. However this was only somewhat effective in reducing ID porosity. Using a melt button with higher liquidus temperature (90Cu-10Ni) increased the melt superheat, but still did not eliminate porosity. A porous layer extending 4-10mm from the substrate was found in these preforms with a total wall thickness of 25-30mm. Further improvements may be possible by increasing the superheat even more and using an insulating fiber substrate. Fully dense CA150 zirconium copper preforms are difficult to obtain, largely because of the high thermal diffusivity and effective singular melting point. These properties prevent thermal gradients and mushy zones from forming which are necessary for an incremental solidification process such as spray deposition.

Chemistries of the as-sprayed material, starting feedstock, and the SAE specification for ZrCu are listed in Table 4. The feedstock was rich in zirconium at 0.22 w/o and losses during deposition brought down the level to the equilibrium solid solution limit (0.15w/o). Compared to the feedstock oxygen content doubled to 70 - 90ppm, still a reasonable level for this alloy. Excessive oxygen ties up the zirconium and limits precipitation hardening. Nitrogen pickup was insignificant.

A plot of Rockwell F hardness verses aging temperature is shown in Figure 9. The cold worked feedstock had a hardness of 88, while the as-sprayed material had a much lower hardness as would be expected in a slow cooled condition. Solution treating brought the hardness levels in line with the as-sprayed material (33 HRF). Response was similar for both, with peak hardness at an aging temperature of 500°C. Lower peak hardness of the spray formed material can be attributed to a decrease in zirconium content.

Tensile Results of ZrCu

Hardness response for the four heat treatments are given in Table 3. These follow the same basic pattern as the tensile results. It is not clear why the hardness values are higher for this set of heat treatments than those measured in the previous heat treatment study. Hardness measurements were redone for both sets and were found to be repeatable. Cold working during machining of the tensile specimens may be one possible cause.

Tensile test results for the five groups are given in Table 5. The highest strengths were seen in the as-sprayed material without heat treating. A slight decrease in properties were seen with the solution treated & aged material, but the values are still in line with typical wrought + solution treated & aged product [2]. High strength without hot working is a good indication of the density and fine microstructure that are generated during spray forming. Elongation to fracture was also in line with reported data, although the reduction in area was slightly less.

With higher solution temperature and time (group 4 & 5), strength decreases. This can be mainly attributed to increased grain size. At a test temperature of 540°C (1000°F) yield strength and reduction in area were slightly below the wrought properties, however ultimate strength and elongation were comparable.

CONCLUSIONS

Thermal Response Study

Liner thermal response (melting) is dependent upon section thickness, geometry, and duration of spray. Melting of the copper liner did provide intimate contact, a metallurgical bond, and aided in filling ID porosity in the alloy 625 jacket. From the small test pieces used in this study, it does not appear that excess melting and damage to liner cooling passages will occur in the full scale part. Active cooling using the channels can be used to prevent melting problems.

Interlayer Braze Study

The results for the braze interlayer experiments were encouraging. With the application of an oxide free interlayer it should be possible to achieve a metallurgical bond and simultaneously fill porosity at the interface. The braze layer must be thermally bonded to the liner and liner must have sufficient thermal mass to prevent braze melting during deposition. Effecting the bond by brazing must be performed in a post-deposition heat treatment. Aluminum as an interlayer material did form a metallurgical bond with the copper and alloy 625, however oxides introduced during plasma spray weakened the joint. Similar weakening of the joint was seen using a copper manganese nickel braze.

Spray Forming of ZrCu Liner

Zirconium copper is susceptible to ID "cold" porosity; this may be due to its high thermal conductivity and narrow effective freezing range. Porosity may be reduced by the use of low thermal mass fiber mandrels and adjustment of process parameters (mainly increased melt superheat). Outside the ID material, fully dense material was achieved with minimal changes in nitrogen, oxygen, and zirconium levels. Similar hardness response to aging was seen in the spray formed product and the wrought feedstock. With the exception of reduction in area and yield strength at 540°C (1000°F), tensile properties were closely in line with wrought properties; a strong indication of the fine microstructure and density obtained by spray forming.

ACKNOWLEDGEMENTS

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REFERENCES

1. Moran, A.M., Palko, W., Journal of Metals, p 12-15, December 1988.
2. Aerojet Technical Data from Dr. Thinh Nguyentat, November 1990.

Table 1. Thermal Response Study Process Parameters

Run #	Liner Thick. (mm)	Liner Material	Deposition Rate (kg/m)	Jacket Thick. (mm)	Gas/Metal Ratio (kg/kg)
98	1.5	CA122	17.3	11	.74
115	3.6	CA101	22.4	15	.51
102	12.7	CA101	17.6	11	.68

Table 2. ZrCu Liner Spray Parameters

Run #	Deposition Rate (kg/m)	Gas/Metal Ratio (kg/kg)	Melt Button Material	Scan Angles	Exhaust Temp (°C)	Preform Temp (°C)
111	25.2	0.51	ZrCu	0	207	1154
112	31.2	0.41	ZrCu	0	231	1141
113	28.1	0.44	ZrCu	-1/-6°	238	1144
114	32.0	0.36	90-10CuNi	-1/-6°	257	1148
117	47.3	0.26	90-10CuNi	-1/-6°	290	1121

Table 3. Tensile Test Heat Treatment Schedule & Hardness

Group	Heat Treatment	Tensile Test Temperature	Hardness Rockwell F
1.	As-Sprayed	Room Temp. (RT)	62.8
2.	As-Sprayed + 1700F/1hr/furnace cool (FC) to RT + 1100F/1hr/FC	RT	55.7
3.	Same as Group #2.	1000°F in argon	Same as Group #2
4.	As-Sprayed + 1700F/4hr/FC + 1100F/1hr/FC	RT	53.8
5.	As-Sprayed + 1800F/4hr/FC + 1100F/1hr/FC	RT	60.9

Table 4. Chemical Analysis of ZrCu Preforms
(weight percent)

Material	Copper	Zirconium	Oxygen	Nitrogen
Preform #111	99.7	0.16	0.009	0.001
Preform #114	99.8	0.15	0.007	0.002
Feedstock	99.7	0.22	0.004	<0.001
SAE J463 Specification	>99.8	0.10-0.20	NA	NA

Table 5. ZrCu Tensile Test Report

TENSION TEST RESULTS FOR .252" DIA ROUND TENSILES.

SPEC.ID	TEST TEMP	UTS KSI	UTS MPa	.2%YS KSI	YS MPa	%EL	%RA	YS/UTS KSI	B/S KSI	EF ¹	FRAC TYPE
1A	RT	33.3	230	13.6	94	40	54	0.41	1.0	0.77	NO FAILURE
1B	RT	32.9	227	12.8	88	42	53	0.39	*	0.76	
1C	RT	33.1	228	13.2	91	46	59	0.40	*	0.90	
1D	RT	32.9	227	11.7	81	44	53	0.36	1.3	0.76	
1E	RT	33.2	229	13.0	90	51	66	0.39	*	1.08	
AVERAGE		33.1	228	12.9	89	45	57	0.39	1.1	0.85	
2A	RT	31.4	217	12.6	87	38	59	0.40	*	0.90	NO FAILURE
2B	RT	32.4	223	12.3	85	45	65	0.38	*	1.06	
2C	RT	32.2	222	12.4	85	45	47	0.38	*	0.64	
2D	RT	32.3	223	12.4	86	41	62	0.39	*	0.96	
AVERAGE		32.1	221	12.4	86	42	58	0.39	*	0.89	
3A	1000	12.1	83	5.2	36	39	54	0.43	*	0.77	
3B	1000	13.6	94	5.8	40	85	69	0.43	*	1.16	
3C	1000	12.4	86	6.6	46	33	59	0.53	*	0.89	
3D	1000	14.2	98	7.1	49	72	64	0.50	*	1.02	
AVERAGE		13.1	90	6.2	43	57	61	0.47	*	0.96	
4A	RT	31.1	214	10.1	70	39	62	0.33	0.0	0.96	
4B	RT	28.6	197	11.1	76	25	37	0.39	0.8	0.45	
4C	RT	29.8	205	10.3	71	32	49	0.35	1.3	0.66	
4D	RT	29.8	205	10.0	69	31	49	0.34	*		
4E	RT	30.2	208	10.3	71	30	49	0.34	*		
AVERAGE		29.9	206	10.4	71	32	49	0.35	0.7	0.4	
5A	RT	29.8	206	10.7	73	40	60	0.36	*	0.93	**
5B	RT	27.7	191	8.9	61	29	64	0.32	*	1.03	
5C	RT	30.5	210	9.2	63	47	59	0.30	*	0.90	
AVERAGE		29.3	202	9.6	66	39	61	0.33	*	0.95	

SPECIMENS 2A-2D WERE STRAIN GAGED AND ATTACHED WITH 1" EXTENSOMETER
SPECIMENS 1A-1E, 4A-4E, AND 5A-5C WERE ATTACHED WITH 1" EXTENSOMETER

* NO BREAK LOAD MEASUREABLE

** .242 DIA, FAILED AT KNIFE EDGE OF EXTENSOMETER

¹ True plastic strain at fracture

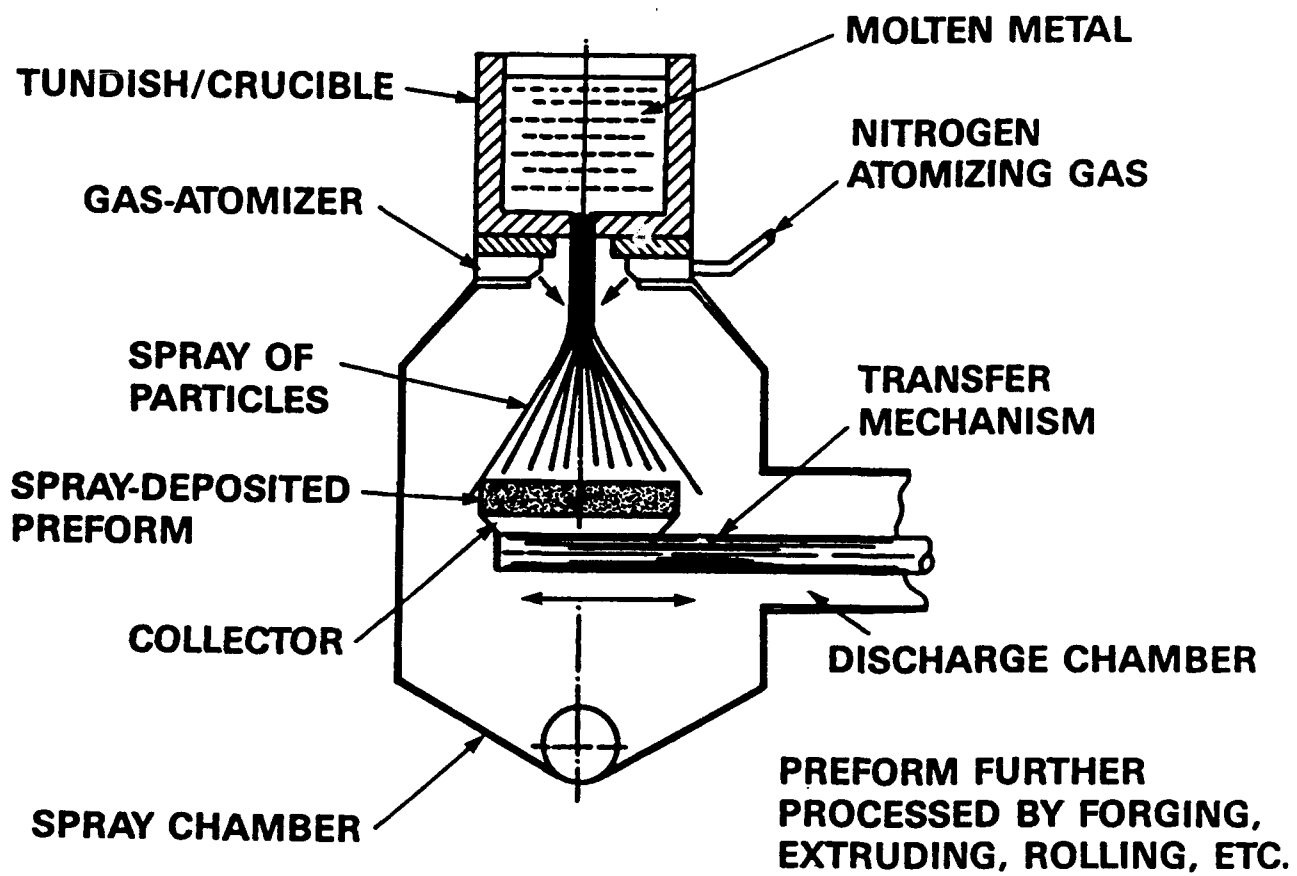


Figure 1. Schematic of the spray deposition process.

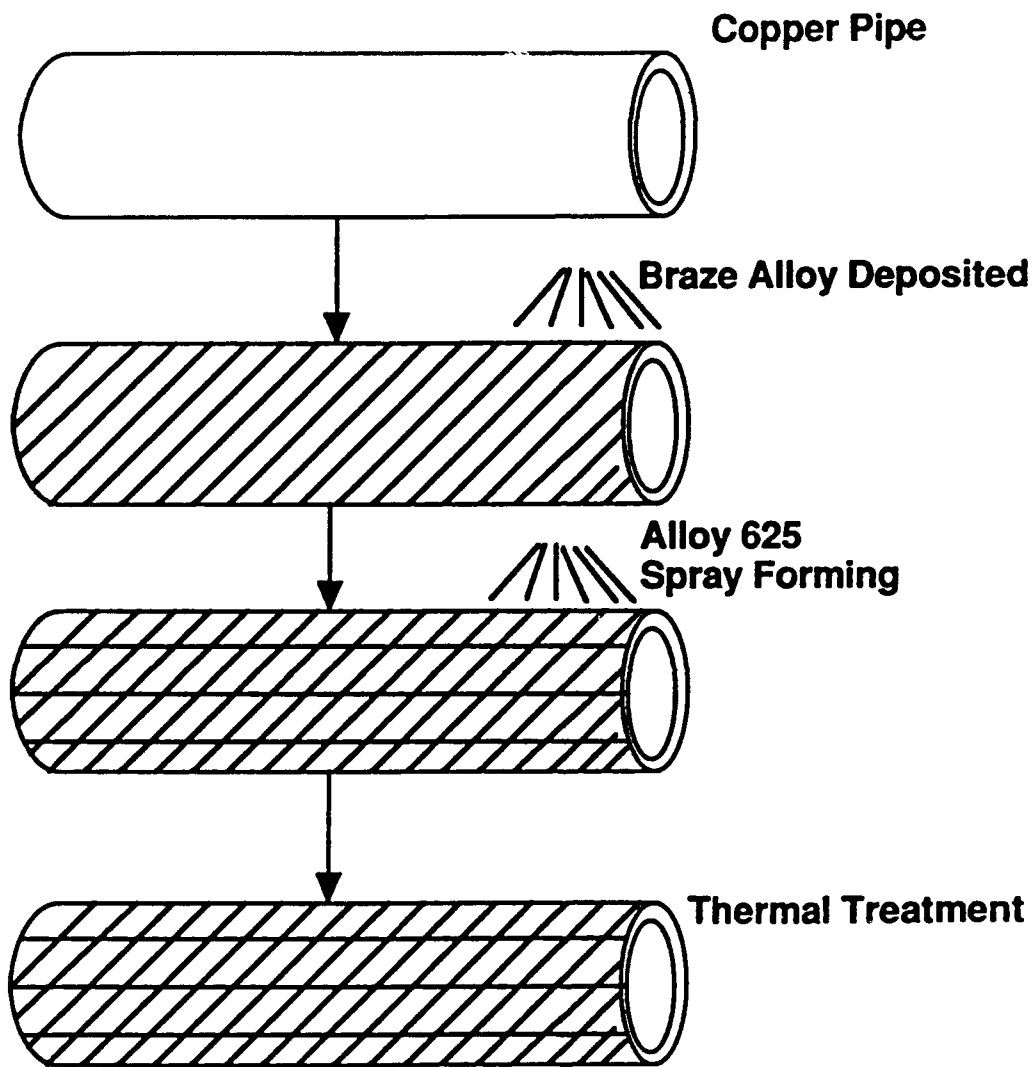


Figure 2. Schematic of interlayer braze processing.

Point A



Alloy 625

Interface

Copper

Point B

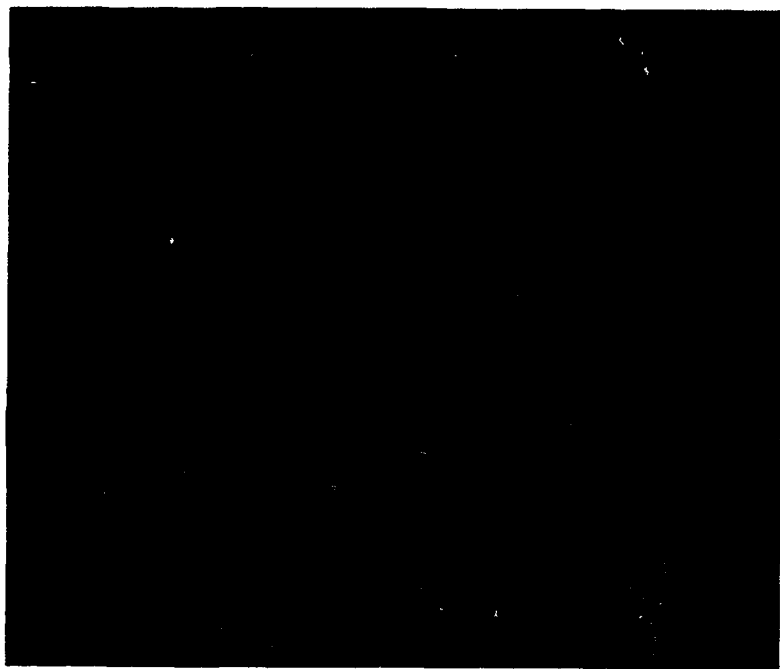


Figure 3. Micrographs of alloy 625/copper interface. Point A was from midlength of tubular, point B was from the start end of the tubular.

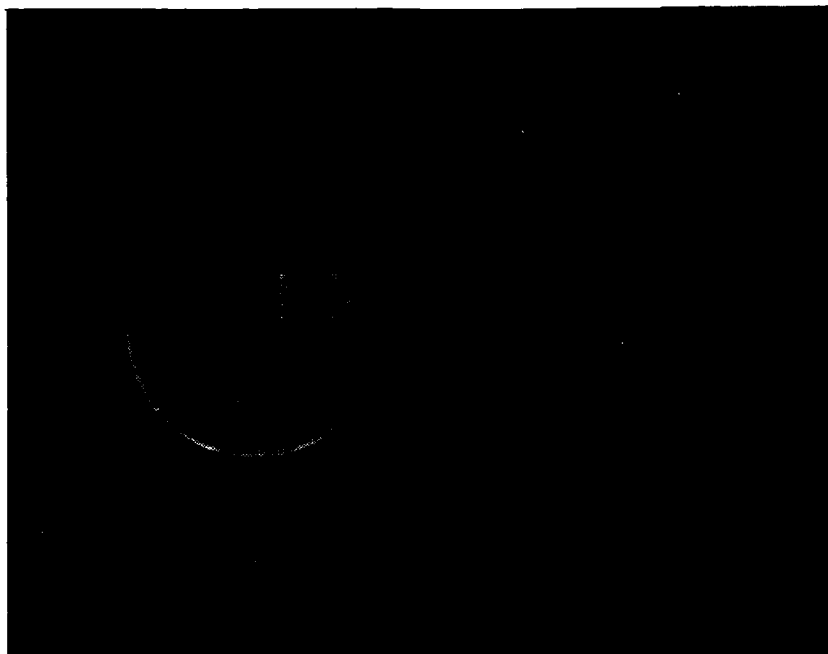
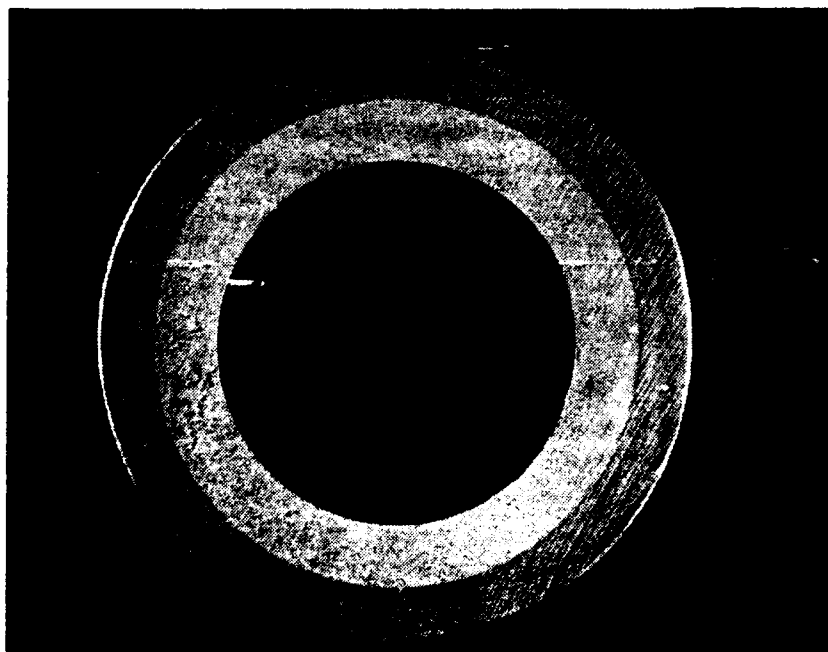


Figure 4. Alloy 625 jacket spray formed onto 12mm wall copper liner.



- ALLOY 625

- ALUMINUM

- COPPER TUBE

Figure 5. Micrograph of alloy 625/copper interface with plasma sprayed aluminum interlayer.

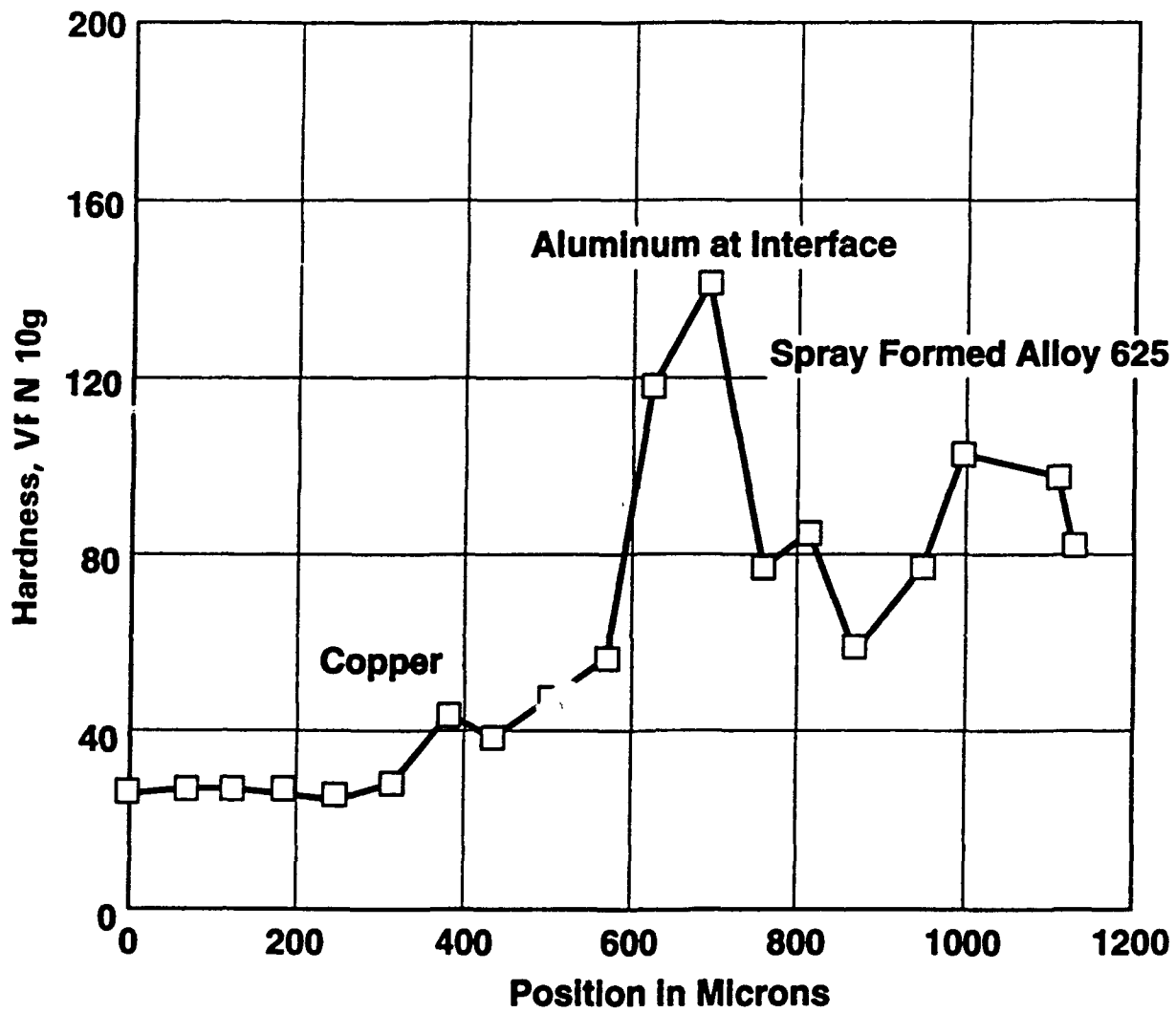
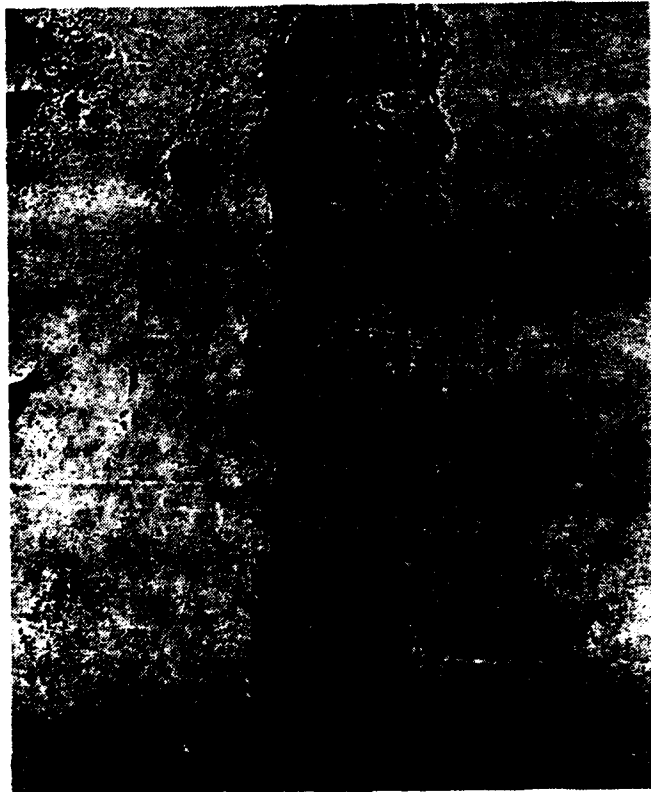
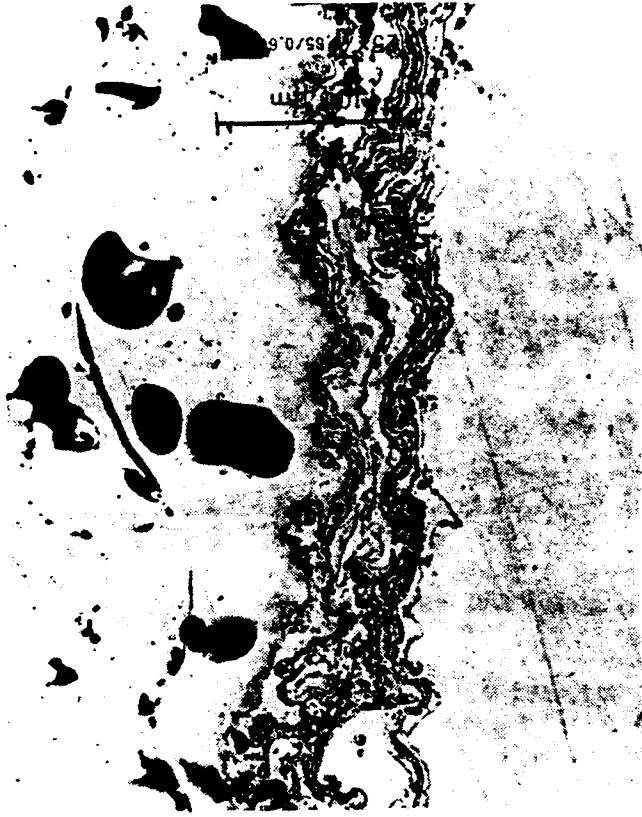


Figure 6. Microhardness profile of alloy 625/copper interface with plasma sprayed aluminum interlayer.



Before Heat Treat



After Heat Treat

Spray
Formed
Alloy 625

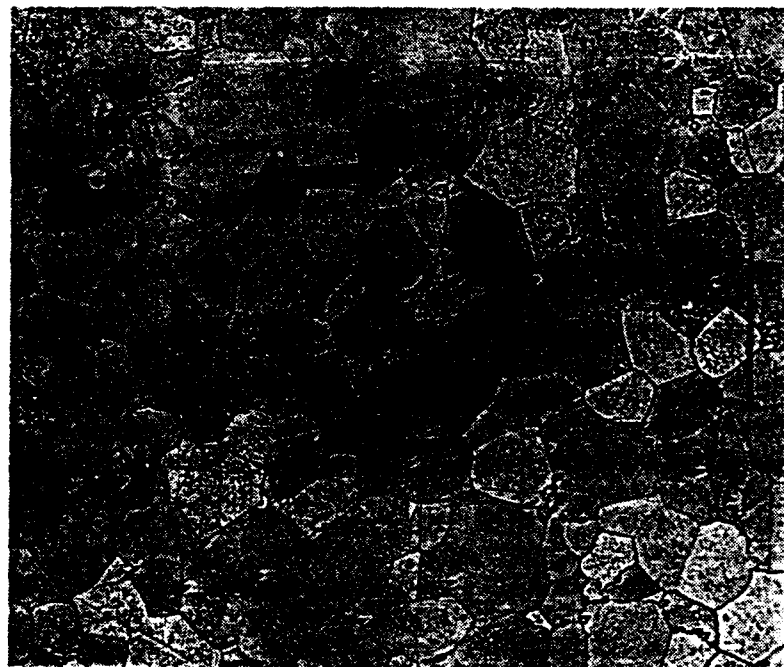
CuMnNi
Interface

Copper

Figure 7. Micrographs of alloy 625/copper interface with HVOF sprayed Cu-Mn-Ni braze interlayer, before and after heat treatment.



WROUGHT



SPRAY FORMED

Figure 8. Micrographs of wrought and spray formed ZrCu alloy. Alcoholic ferric chloride etch.

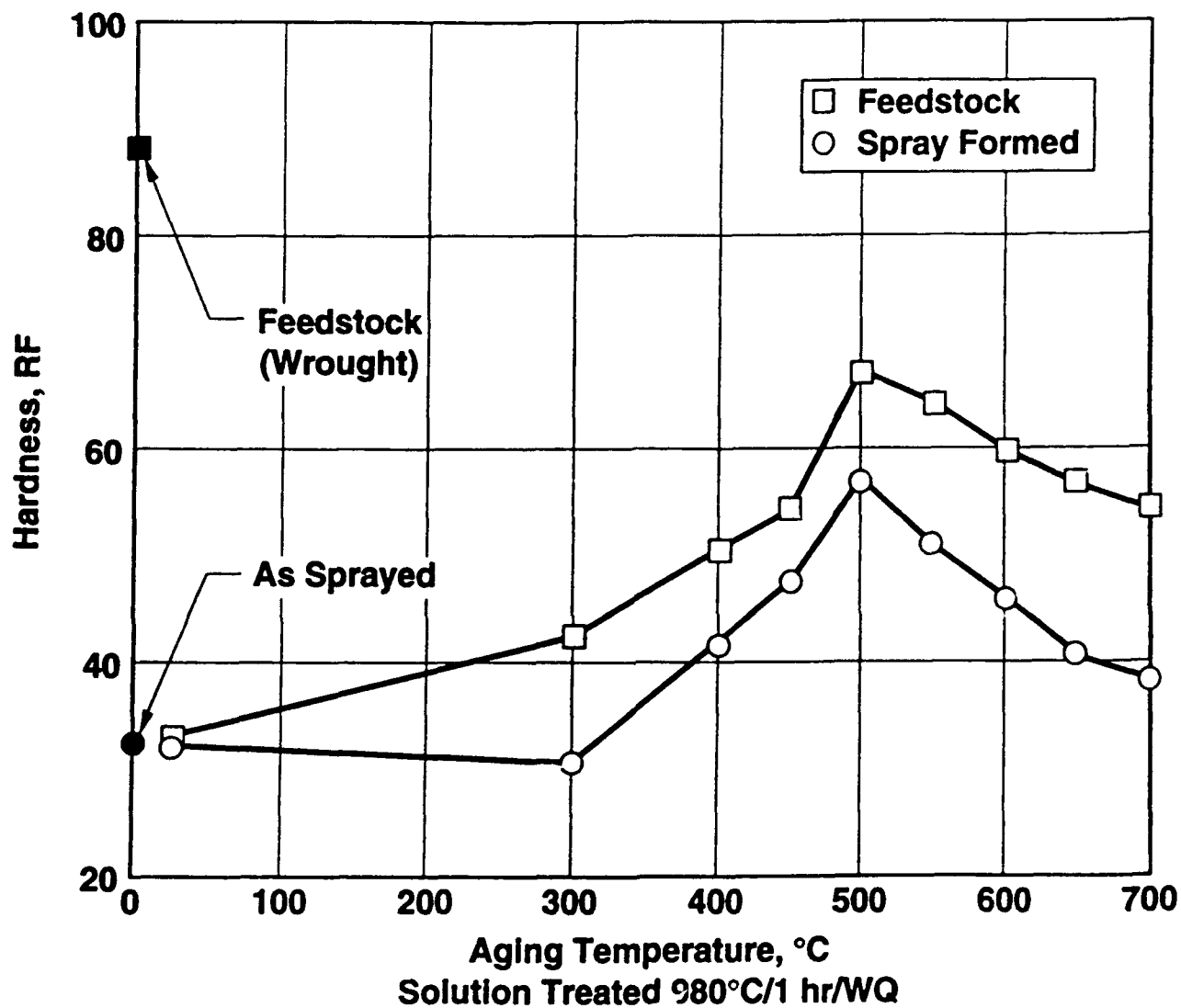


Figure 9. Hardness response of spray formed and feedstock ZrCu alloy as a function of aging temperature.

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13. ABSTRACT (Maximum 200 words) High deposition rate spray forming is a new technology which has been developed to reduce the costs and to improve the performance of a wide variety of engineering alloys. The purpose of this investigation was to demonstrate the ability to produce layered bimetallic structures via the spray forming process. Copper tubulars were used as collectors for spray deposited alloy 625. The collectors were varied in thickness and various interfacial alloys were incorporated to promote bonding between the copper and superalloy. Microstructures and bond strength were determined.				
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